

## Studies of Water-in-Oil Emulsions: Energy Threshold of Emulsion Formation

Merv Fingas and Ben Fieldhouse  
Emergencies Science Division  
Environmental Technology Centre  
Environment Canada  
Ottawa, Ontario  
E mail: Fingas.Merv@etc.ec.gc.ca

Joe Mullin  
U.S. Minerals Management Service  
Herndon, Virginia

### ABSTRACT

This paper summarizes studies to determine the energy onset of water-in-oil formation. The total energy applied to the oil/water in the emulsion formation apparatus was varied from a minimum to a maximum value of approximately 200 to 600,000 ergs (equivalent to  $2 \times 10^{-5}$  to  $6 \times 10^{-2}$  Joules). Turbulent energy, which is largely responsible for emulsion formation, could not be measured.

Four, clearly-defined states of water-in-oil have been characterized by a number of measurements and by their visual appearance, both on the day of formation and one week later. This study demonstrated that the energy threshold for the onset of the two states known as stable emulsion and entrained water, is usually very low, 300 to about 1500 ergs, corresponding to a rotational rate in the formation apparatus of about 1 to 3 rpm. It was shown that for the one oil type, Bunker C, which forms an entrained water state, that there is no increase in stability with increasing energy input, after the initial formation point. The oil that forms a meso-stable emulsion, Prudhoe Bay, showed a similar tendency in that after the energy onset, which occurs at a high level of about 25,000 ergs, there is no apparent increase in stability. Both oils that form stable emulsions, Arabian Light and Sockeye showed an increasing stability with increasing energy, although the rate of increase was gradual with increasing energy.

### 1.0 Introduction

Studies in the past four years by Environment Canada have shown that a class of very 'stable' emulsions exists, characterized by their persistence over several months (Fingas *et al.*, 1995, 1996, 1997, 1998). These stable emulsions actually undergo an increase in viscosity over time. Monitoring of these emulsions has been performed for as long as 3 years in the laboratory. 'Unstable' emulsions do not show this viscosity increase and their viscosity is less than about 20 times greater than the starting oil. The viscosity increase for stable emulsions is at least three orders-of-magnitude greater than the starting oil. It was concluded both on the basis of the literature and experimental evidence above, that certain emulsions can be classed as stable (Sjöblom and Førdedal, 1996; Førdedal *et al.* 1996a, 1996b; Førdedal and Sjöblom, 1996; Dukhin and Sjöblom, 1996). Some (if not all or many) stable emulsions increase in apparent viscosity with time (ie. their elasticity increases). The stability derives from the strong visco-elastic interface caused by asphaltenes, perhaps



along with resins (do Carmo Marques *et al.* 1997; Friberg and Yang, 1996; McLean and Kilpatrick, 1997a, 1997b; Neumann and Paczynska-Lahme, 1996; Puskas *et al.* 1996). Increasing viscosity may be caused by increasing alignment of asphaltenes at the oil-water interface.

Mesostable emulsions are emulsions that have properties between stable and unstable emulsions (really oil/water mixtures) (Fingas *et al.* 1995). It was suspected that mesostable emulsions lack sufficient asphaltenes to render them completely stable or still contain too many de-stabilizing materials such as aromatics. Further light is shed on their stability in this paper. The viscosity of the oil may be high enough to stabilize some water droplets for a period of time. Mesostable emulsions may degrade to form layers of oil and stable emulsions. Mesostable emulsions can be red in appearance or black. Mesostable emulsions are probably the most commonly-formed emulsions in the field.

Unstable emulsions are those that decompose (largely) to water and oil rapidly after mixing, generally within a few hours. Some water (usually less than about 10%) may be retained by the oil, especially if the oil is viscous.

The most important measurements to characterize emulsions are forced oscillation rheometry studies. The presence of significant elasticity clearly defines whether or not a stable emulsion has been formed. The viscosity by itself can be an indicator (not necessarily conclusive, unless one is fully certain of the starting oil viscosity) of the stability of the emulsion. Colour is an indicator, but may not be definitive. Water content is not an indicator of stability and is error-prone because of 'excess' water that may be present. It should be noted however that stable emulsions have water contents greater than 70% and that unstable emulsions or entrained water-in-oil generally have water contents less than 50%. Water content after a period of about one week is found to be more reliable than immediate water content. This is because separation will occur in those emulsions that are less stable.

An important aspect of emulsions that has not been studied to date is the kinetics of emulsion formation and the energy levels associated with the formation of emulsions. Such information is needed to understand the emulsification process and to model the process. This paper reports on initial experiments to examine the kinetics and the formation energy of emulsions. It is important to note that turbulent energy is felt to be the most important form of energy related to emulsion formation. Turbulent energy could not be measured in this apparatus, so the total energy was used as an estimate of the energy available for emulsion formation.

## **2.0 Experimental**

Water-in-oil emulsions were made in a rotary agitator and then the rheometric characteristics of these emulsions studied over time. Oils were taken from the storage facilities at the Emergencies Science Division. Properties of these oils are given in standard references (Jokuty *et al.* 1996).

This paper reports on two series of experiments, one to measure the energy threshold and a second smaller set of experiments made only with Sockeye oil to measure the variance in rheological measurements associated with such experiments. The energy threshold measurements were conducted by varying the rotational rate, and hence the energy of the apparatus used to make the emulsions. Analysis of the emulsions was conducted using rheological measurements as described herein and standard visual observations.



**Emulsion Formation** - Emulsions were made in an end-over-end rotary mixer (Associated Design). The apparatus was located in a temperature controlled cold room at a constant 15 degrees Celsius. The mixing vessels were 2.2 L FLPE wide-mouthed bottles (Nalge). The mixing vessels were approximately one-quarter full, with 600 mL salt water (3.3% w/v NaCl) and 30 mL of the sample crude oil or petroleum product. The vessels were mounted into the rotary mixer, and allowed to stand for several hours (usually three) to thermally equilibrate. The vessels were then rotated for 12 hours at a rate between 1 and 55 rpm. The resulting emulsions were then collected into Fleaker jars, covered, and stored in the same 15 degree cold room. Analysis was performed on the day of collection a short time after formation.

**Rheology** - The following apparatuses were used for rheological analysis: Haake RS100 RheoStress rheometer, IBM-compatible PC with RheoStress RS Ver. 2.10 P software, 35 mm and 60 mm parallel plates with corresponding base plates, clean air supply at 40 p.s.i., and a circulating bath maintained at 15.0 degrees Celsius. Analysis was performed on a sample spread onto the base plate and raised to 2.00 mm from the measuring plate, with the excess removed using a teflon spatula. This was left for 15 minutes to thermally equilibrate at 15 degrees Celsius.

For characterization of apparent viscosity, the cup and spindle system was used. This consisted of the Haake Roto visco RV20 with M5 measuring system, Haake Rheocontroller RC20 and PC with dedicated software package Roto Visco 2.2. The sensors and vessels used were the SVI spindle and SV cup. The shear rate was one reciprocal second. The viscometer was operated with the following ramp times: one minute to target shear rate 1/s; one minute at target shear rate (1/s). The temperature was maintained at 15 degrees Celsius. Fifteen minutes was allowed for the sample to thermally equilibrate.

**Forced Oscillation** - A stress sweep at a frequency of 1 reciprocal second was performed first to determine the linear viscoelastic range (stress independent region) for frequency analysis. This also provides values for the complex modulus, the elasticity and viscosity moduli, the low shear dynamic viscosity, and the  $\tan(\delta)$  value. A frequency sweep was then performed at a stress value within the linear viscoelastic range, ranging from 0.04 to 40 Hz. This provides the data for analysis to determine the constants of the Ostwald-de-Waele equation for the emulsion.

**Viscosities** - The apparent dynamic viscosity was determined on the plate-plate apparatus as well, and corrected for their non-Newtonian behaviour using the Weissenberg equation. A shear rate of 1 reciprocal second was employed for a period of one minute, without ramping.

**Water Content** - A Metrohm 701 KF Titrino Karl-Fischer volumetric titrator and Metrohm 703 Ti Stand were used. The reagent was Aquastar Comp 5 and the solvent, 1:1:2 Methanol:Chloroform:Toluene. The specific method used was as follows: standardize the titre and blank the solvent. Stir the sample emulsion to get a relatively homogeneous mixture. Fill a 1 mL plastic syringe with emulsion, trying to avoid free water pockets present in the sample. Eject all but 0.1 mL; this should remove most of the free water from the more viscous emulsion. Weigh the sample syringe and inject into the reaction vessel, being careful the sample goes into the solution and not onto the vessel walls. Reweigh the syringe and enter the difference into the titrator. Initiate titration. Weight percentage of water is displayed.



### 3.0 Energy Calculations

The general layout of the rotational device is shown in Figure 1.

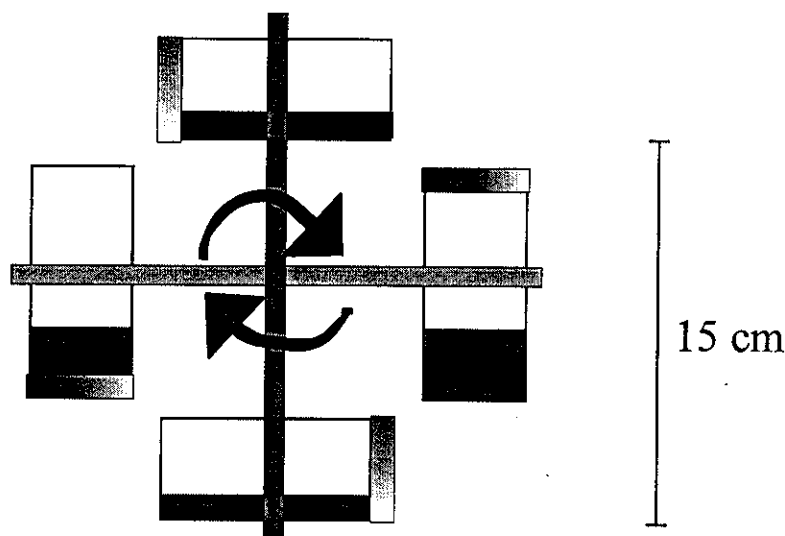


Figure 1 Diagram of the Emulsion Formation Device

The simple way to calculate the total energy exerted on the oil/water in the device is to calculate the total kinetic energy of the system.

The total kinetic energy in each bottle is given by:

$$KE = \frac{1}{2} MV^2 \quad (1)$$

Where: KE is the energy in ergs

M is the mass in grams, here approximately 620 g of water and oil

V is the velocity in cm/s which is  $2\pi r$  - which is  $\text{rpm}/60 \times 7.5 \text{ cm}$

Kinetic energy by this formula is then  $196 \times \text{rpm}^2$  ergs. Ergs were used in this study because they are a much more convenient unit than the SIU Joules at these low energy levels. This simple formulation will be used to assign an energy level to each rotational velocity. Again, it is important to note that the energy estimated here is the total energy input to the system, and not turbulent energy which is the prime factor in emulsion formation.

### 4.0 Results and Discussion

The rheological data associated with the energy threshold experiments are given in Table 1. The second column of Table 1 is the rotational rate of the formation vessel. The third column is the calculated kinetic energy applied to the system in ergs. The fourth column is the complex modulus which is the vector sum of the viscosity and elasticity. The fifth column shows stability of the emulsion which is the complex modulus divided by the starting oil viscosity (Fingas *et al.* 1998). The sixth column gives the water content of the emulsion. The seventh column gives the assessment of the stability of the emulsion based on both visual appearance and rheological properties. The eighth and ninth columns gives the viscosity of the emulsions. The eighth column is the viscosity as given by the RV-20 instrument and





Table 1 Experimental Parameters and Results

Oil	RPM	Energy ergs*	Complex Modulus mPa	Stability s <sup>-1</sup>	H <sub>2</sub> O (w/w)	Visual Stability	Oil Viscosity	
							RV20	RS100 mPa.s
Arabian Light	1.0	196		1		Unstable		14
Arabian Light	1.3	331		1		Unstable		14
Arabian Light	2.8	1537		1		Unstable		14
Arabian Light	3.1	1884		1		Unstable		14
Arabian Light	5.1	5098	4.0E+04	2860	84.73	Stable	4.9E+03	5.5E+03
Arabian Light	5.3	5506	8.2E+03	590	81.82	Stable		1.5E+03
Arabian Light	10.3	20794	1.5E+04	1070	85.60	Stable	7.0E+03	4.2E+03
Arabian Light	10.5	21609	2.4E+05	17140	90.31	Stable		1.1E+03
Arabian Light	20.4	81567	2.5E+05	17500	93.22	Stable	5.9E+04	
Arabian Light	21.0	86436	8.3E+04	5930	85.06	Stable		1.2E+04
Arabian Light	29.8	174056	3.1E+05	22140	92.3	Stable		4.0E+04
Arabian Light	29.8	174056	3.3E+05	23570	92.56	Stable	6.0E+04	5.0E+04
Arabian Light	39.8	310472	7.9E+05	56430	91.58	Stable	9.1E+04	
Arabian Light	39.8	310472	6.6E+05	47140	90.73	Stable	1.0E+05	
Arabian Light	40.3	318322	3.0E+05	21070	92.06	Stable	5.9E+04	7.0E+04
Arabian Light	41.8	342459	8.2E+05	58570	90.4	Stable		3.0E+04
Arabian Light	55	592900	9.4E+05	67140	88.71	Stable	1.4E+05	5.0E+04
Bunker C	1.0	196	2.2E+05	1	46.83	Entrained		6.5E+04
Bunker C	1.3	331	2.4E+05	10	49.91	Entrained	9.7E+04	9.0E+04
Bunker C	2.8	1537	3.6E+05	10	58.22	Entrained		1.2E+05
Bunker C	3.1	1884	3.6E+05	10	61.72	Entrained	1.5E+05	1.3E+05
Bunker C	5.1	5098	4.0E+05	10	60.32	Entrained	1.7E+05	1.3E+05
Bunker C	5.3	5506	4.4E+05	10	62.96	Entrained		1.3E+05
Bunker C	10.3	20794	2.9E+05	10	49.91	Entrained	1.2E+05	1.0E+05
Bunker C	10.5	21609	3.6E+05	10	54.71	Entrained		1.3E+05
Bunker C	20.4	81567	5.1E+05	10	64.80	Entrained	1.7E+05	
Bunker C	21.0	86436	4.5E+05	10	54.16	Entrained		1.2E+05
Bunker C	29.8	174056	3.4E+05	10	48.89	Entrained		9.5E+04
Bunker C	29.8	174056	3.5E+05	10	45.82	Entrained	9.1E+04	8.5E+04
Bunker C	40.3	318322	3.8E+05	10	49.18	Entrained	1.1E+05	9.5E+04
Bunker C	41.8	342459	3.9E+05	10	47.46	Entrained		1.1E+05
Bunker C	55	592900	3.9E+05	10	49.27	Entrained	1.1E+05	1.1E+05
Prudhoe Bay	1.0	196		1		Unstable		900
Prudhoe Bay	1.3	331		1		Unstable		900
Prudhoe Bay	2.8	1537		1		Unstable		900
Prudhoe Bay	3.1	1884		1		Unstable		900
Prudhoe Bay	5.1	5098		1		Unstable		900
Prudhoe Bay	5.3	5506		1		Unstable		900
Prudhoe Bay	10.3	20794		1		Unstable		900
Prudhoe Bay	10.5	21609		1		Unstable		900
Prudhoe Bay	20.4	81567		1		Unstable		900
Prudhoe Bay	21.0	86436		1		Unstable		900
Prudhoe Bay	29.8	174056		1		Unstable		900
Prudhoe Bay	29.8	174056		1		Unstable		900
Prudhoe Bay	40.3	318322	8.8E+03	10	28.49	Meso	5.1E+03	
Prudhoe Bay	41.8	342459	8.2E+03	10	72.52	Meso		1.3E+03
Prudhoe Bay	55	592900	4.2E+03	5	55.81	Meso	6.7E+03	4.0E+02
Sockeye	1.0	196		1		Unstable		45
Sockeye	1.3	331	1.4E+04	310	82.39	Stable	7.6E+03	3.0E+04
Sockeye	2.8	1537	1.7E+05	3780	89.45	Stable		2.5E+05
Sockeye	3.1	1884	2.1E+05	4670	90.92	Stable	2.7E+04	2.0E+04
Sockeye	5.1	5098	3.9E+05	8670	92.60	Stable	5.4E+04	7.0E+04
Sockeye	5.3	5506	3.6E+05	8000	92.50	Stable		5.0E+04
Sockeye	10.3	20794	1.5E+06	33330	90.57	Stable	4.0E+05	3.0E+05
Sockeye	10.5	21609	1.8E+06	40000	90.56	Stable		2.8E+05
Sockeye	20.4	81567	8.3E+05	18330	92.03	Stable	2.3E+05	
Sockeye	20.7	83984	1.2E+06	26670	90.51	Stable	2.8E+05	
Sockeye	20.7	83984	7.6E+05	16890	91.23	Stable	2.1E+05	
Sockeye	21.0	86436	8.7E+05	19330	91.31	Stable		1.6E+05
Sockeye	29.8	174056	2.8E+06	62220	89.12	Stable		3.5E+05
Sockeye	29.8	174056	3.0E+06	66670	88.17	Stable	6.6E+05	5.0E+05
Sockeye	40.3	318322	3.6E+06	80000	88.95	Stable	9.2E+05	3.0E+05
Sockeye	41.8	342459	3.4E+06	75560	87.95	Stable		4.0E+05
Sockeye	55	592900	4.1E+06	90000	86.44	Stable	7.6E+05	5.8E+05

\*one erg = 10<sup>-7</sup> Joules



the ninth column gives the viscosity derived from the RS 100 instrument. The eleventh column gives the viscosity of the starting oil as measured by the RV-20 instrument. Differences between the viscosity determined by these two instruments are a result of the differences between the two instruments as well as normal measurement variances.

Observations were made on the appearance of the emulsions and were used to classify the emulsions. All of the stable emulsions appeared to be stable and remained intact over seven days in the laboratory. All of the meso-stable emulsions broke after a few days into water, free oil and emulsion. The time for these emulsions to break down varies from about 1 to 3 days. The emulsion portion of these break-down emulsions appears to be somewhat stable, although separate studies on this portion has not been performed because of the difficulty in separating these portions from the oil and water. All entrained water appeared to have larger suspended water droplets. The appearance of non-stable water in oil was just that, the oil appeared to be unchanged and a water layer was clearly visible.

A second set of experiments was conducted to measure the variance in rheological measurements associated with such experiments. The measurements were taken during one day on the Sockeye emulsion at time intervals of approximately one hour intervals. Table 2 shows that the average standard deviation of a viscosity measurement is  $5.3 \times 10^4$  mPa.s (approximately 6 % relative standard deviation) and for the complex modulus,  $2.5 \times 10^5$  mPa (approximately 7 %). This information is useful in interpreting the emulsion formation data.

The stability and energy of formation are plotted for the four emulsions in Figures 2 to 5. The stability in these figures is the complex modulus divided by the starting oil viscosity (Fingas *et al.* 1998). The latter reference traces the development of this index. In summary, the 'stability', as here defined, was found to be the only single parameter that could be used to describe the emulsions mathematically. Furthermore, stability was found to correlate very highly with other indices related to the formation of emulsions. Figure 2 shows that the onset of stability for Arabian Light oil is rapid and increases at an approximately linear rate. Stability is generally taken as the point at which the stability is approximately 1,000 and this is achieved at a very low energy level corresponding to a rotational rate of about 5 rpm. Figure 3 shows the uptake of water by one sample of Bunker C. The Bunker C takes up water very rapidly between 200 to 300 ergs (1 to 1.3 rpm). After the rapid initial uptake, the stability of the water-oil mixtures remains the same and is typical of entrained water in oil. Figure 4 shows the relationship of stability of Prudhoe Bay with increasing energy. Water uptake is again rapid as in Bunker C, but at a higher energy threshold and a meso-stable emulsion is formed. Figure 4 shows the uptake of water for Sockeye. Water uptake is very rapid at first between the energy levels of 300 to 1500 ergs (1.3 to 2.8 rpm) and after this point stability increases slowly with increasing energy. All four oils show several similar features: initial water uptake occurs very rapidly over a short energy range; the energy threshold for initial water uptake is very low, typically around 300 ergs, except for that of Prudhoe Bay which is about 250,000 ergs; that there is no stability increase for the Bunker C in which the water is entrained and for the Prudhoe Bay which forms a mesostable emulsion; and that there is a slow increase in stability with increasing energy for the oils, Sockeye and Arabian Light, which form stable emulsions. Figure 5 shows all four oils plotted on the same axis. This figure confirms the three trends noted.



Viscosity (mPa.s)	
Sample Number	Repeat Number
	1 2 3 4 5
	Standard Deviation
1	9.10E+05 9.68E+05 8.16E+05 6.93E+05 7.60E+05 1.11E+05
2	7.87E+05 8.86E+05 7.96E+05 7.77E+05 8.02E+05 4.38E+04
3	9.33E+05 8.23E+05 9.26E+05 8.37E+05 7.94E+05 6.29E+04
4	8.04E+05 8.03E+05 8.06E+05 8.15E+05 8.10E+05 4.70E+03
5	8.08E+05 7.77E+05 9.42E+05 8.59E+05 8.59E+05 6.27E+04
6	7.88E+05 7.51E+05 7.56E+05 7.91E+05 8.28E+05 3.10E+04
7	7.58E+05 7.31E+05 7.36E+05 8.26E+05 7.33E+05 4.04E+04
Standard Deviation	2.99E+05 3.00E+05 3.01E+05 2.87E+05 2.85E+05 5.27E+04
	Average Std. Dev.

Complex Modulus (mPa)	
Sample Number	Repeat Number
	1 2 3 4 5
	Standard Deviation
1	3.81E+06 3.83E+06 4.07E+06 4.00E+06 3.38E+06 2.69E+05
2	3.95E+06 3.38E+06 3.94E+06 N/M 3.55E+06 2.86E+05
3	4.17E+06 3.46E+06 3.76E+06 3.43E+06 3.51E+06 3.10E+05
4	4.18E+06 4.05E+06 3.75E+06 3.50E+06 3.51E+06 3.10E+05
5	4.03E+06 3.65E+06 3.70E+06 3.74E+06 3.97E+06 1.71E+05
6	3.81E+06 3.86E+06 3.93E+06 3.94E+06 3.51E+06 1.76E+05
7	4.05E+06 4.06E+06 3.37E+06 3.93E+06 3.60E+06 3.05E+05
Standard Deviation	1.42E+06 1.35E+06 1.36E+06 1.44E+06 1.28E+06 2.54E+05 Average Std. Dev.



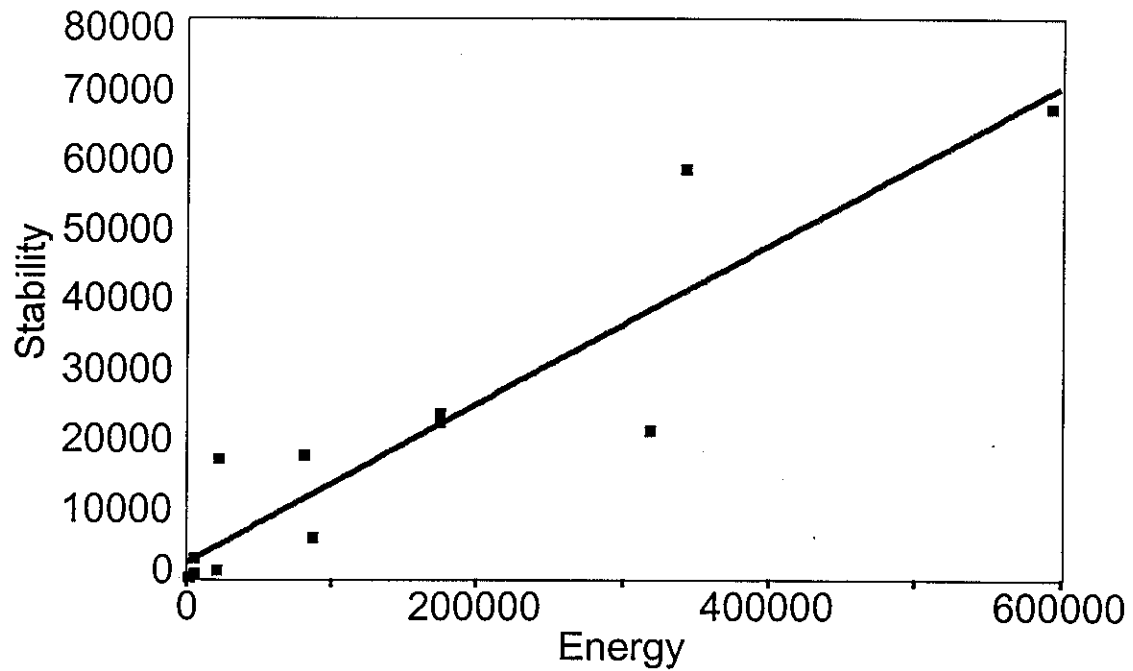


Figure 2 Relation of Stability and Energy of Formation for Emulsion from Arabian Light - Emulsion is Stable

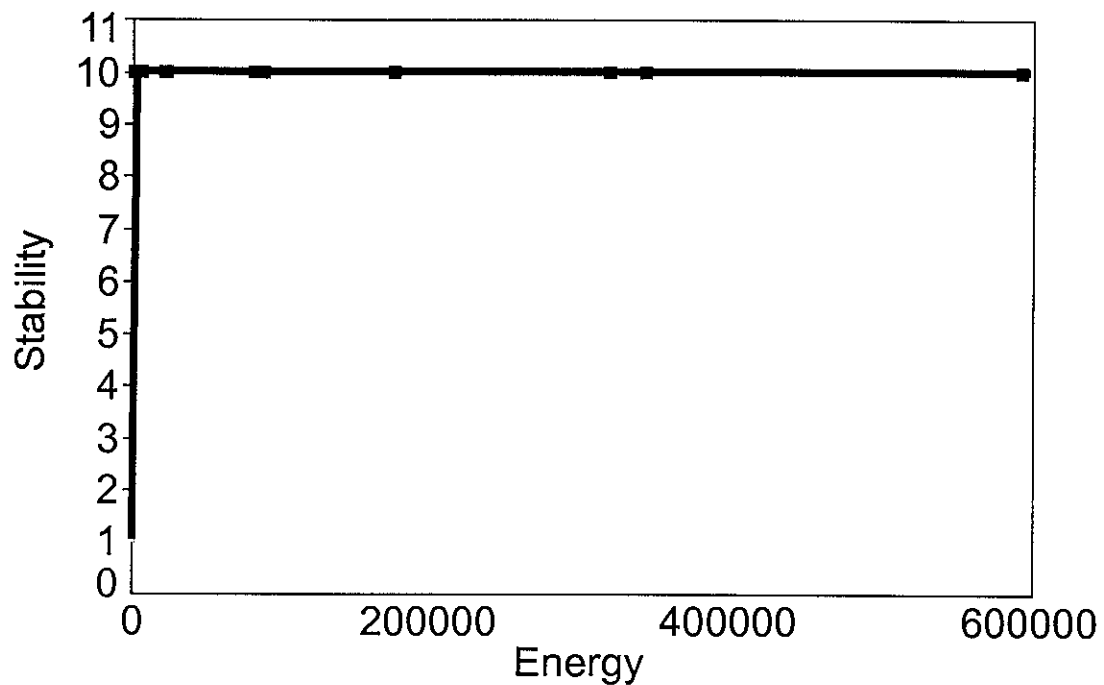


Figure 3 Relation of Stability and Energy of Formation for Water Inclusion of Bunker C - Water is Entrained





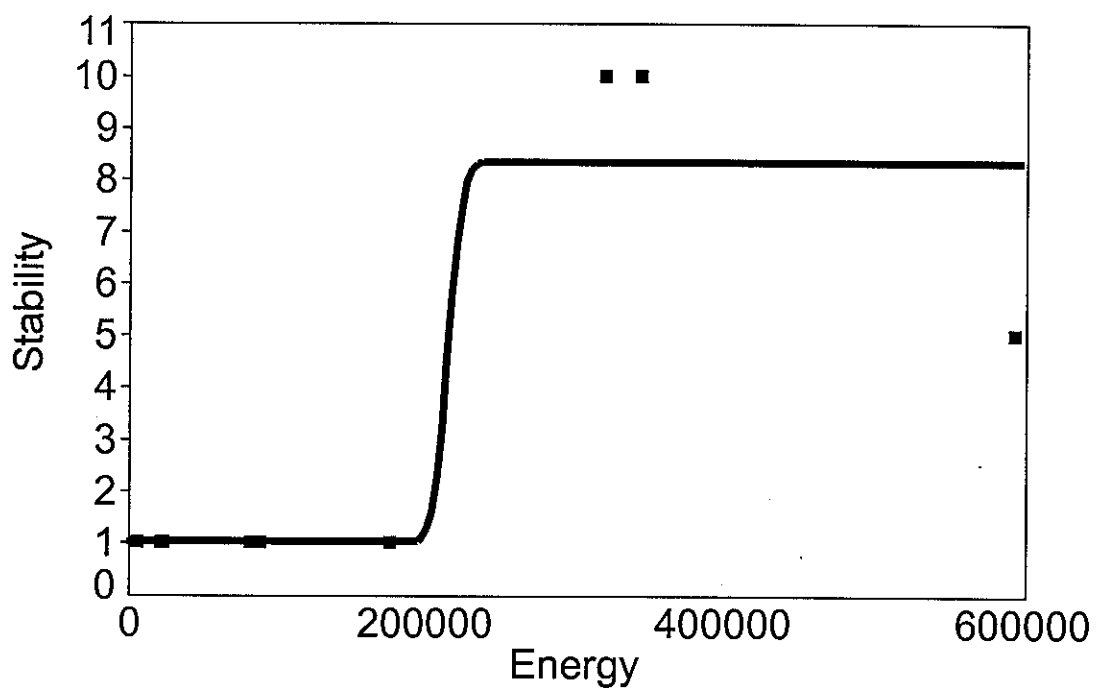


Figure 4 Relation of Stability and Energy of Formation  
Prudhoe Bay - Emulsion is Unstable then Mesostable

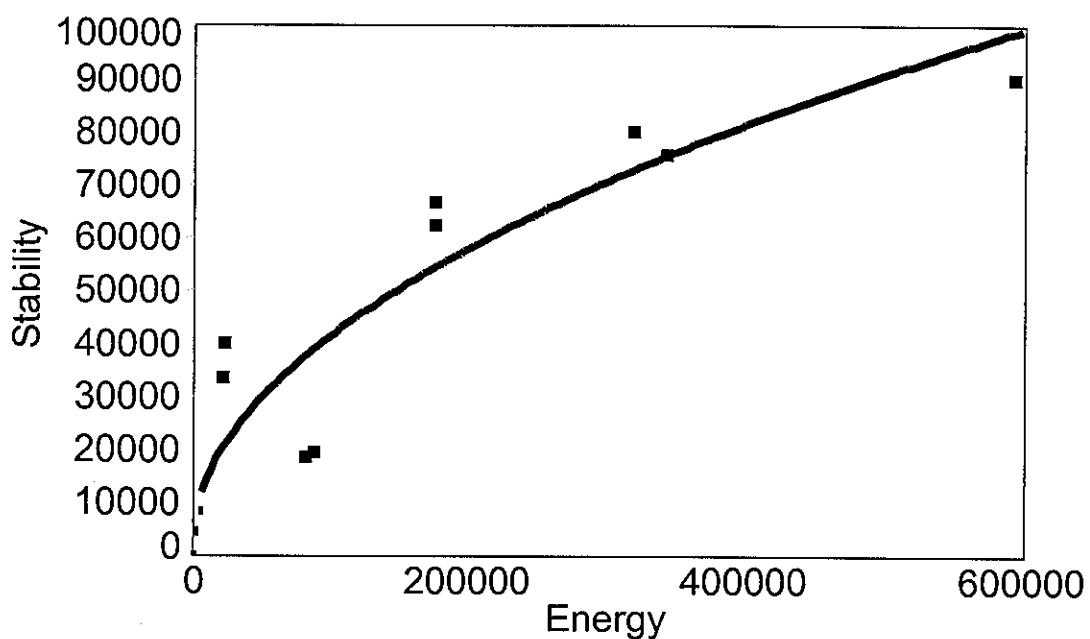


Figure 5 Relation of Stability and Energy of Formation  
for Emulsion from Sockeye - Emulsion is Stable



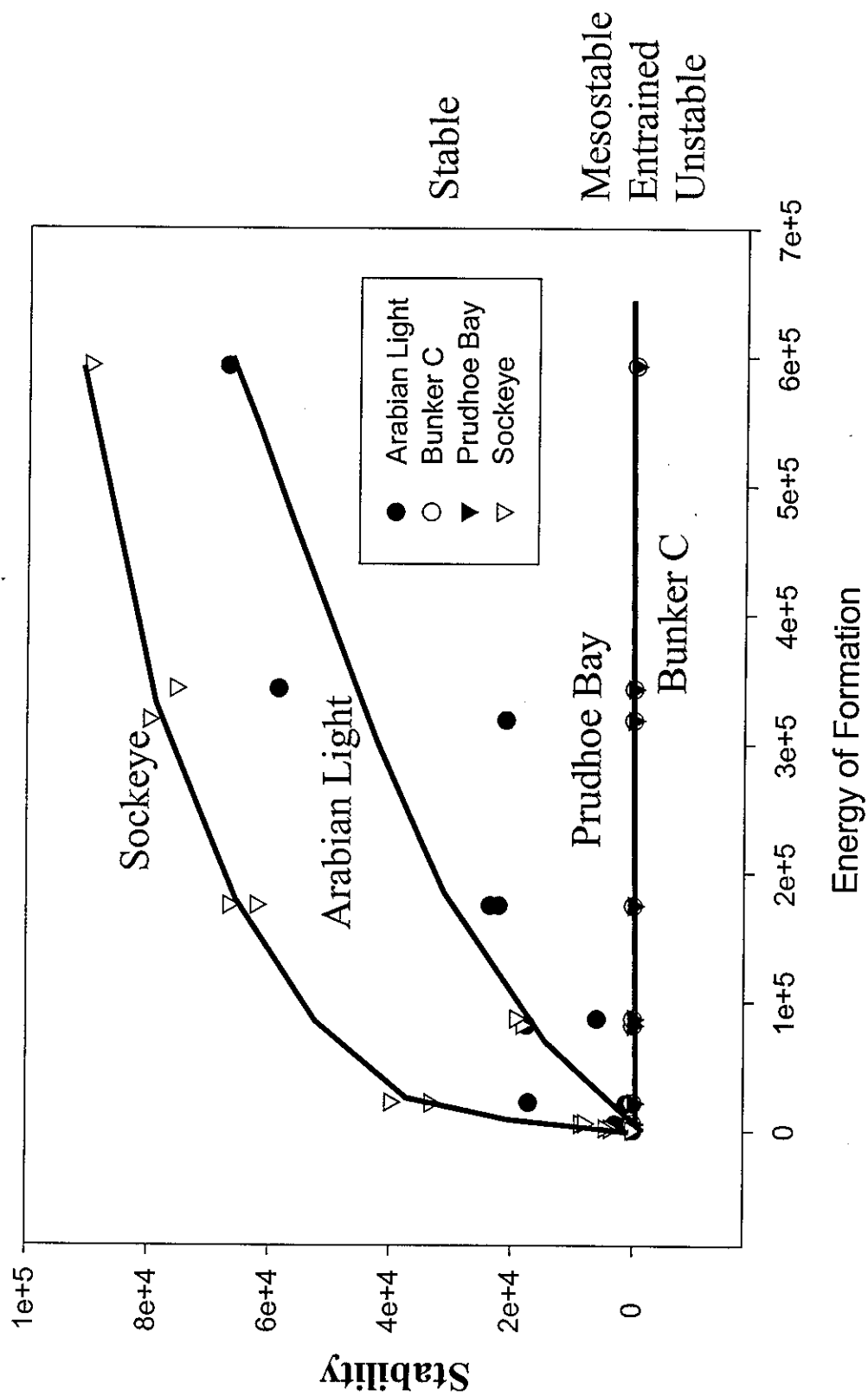


Figure 6 Relation of Stability and Energy of Formation for Emulsion for Four Oils



## 5.0 Conclusions

Four, clearly-defined states of water-in-oil have been shown to be defined by a number of measurements and by their visual appearance, both on the day of formation and one week later (Fingas *et al.* 1998). This study shows that the energy to the onset of the three states known as stable, meso-stable and entrained water, is very low, 300 to about 1500 ergs, corresponding to a rotational rate in the formation apparatus of about 1 to 3 rpm. Total energy applied to the system was used as an indicator value. Turbulent energy, could not be measured. This study also shows that for the one oil type, Bunker C, which forms an entrained water state, that there is no increase in stability with increasing energy input, after the initial formation point. The oil that forms a meso-stable emulsion, Prudhoe Bay, shows a similar tendency in that after the energy onset, which occurs at a high level of about 25,000 ergs, there is no apparent increase in stability. Both oils that form stable emulsions, Arabian Light and Sockeye show an increasing stability with increasing energy, although the rate of increase is gradual with increasing energy.

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